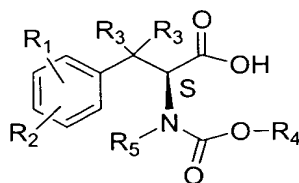


We claim:

1. A process for the preparation of a compound of Formula I:



Formula I

wherein:

R_1 and R_2 are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

R_3 is straight chain alkyl of 1 to 4 carbon atoms;

R_4 is straight chain alkyl of 1 to 4 carbon atoms and branched chain alkyl of 3 to 4 carbon atoms;

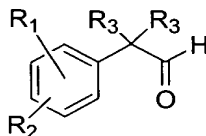
R_5 is straight chain alkyl of 1 to 4 carbon atoms;

comprising:

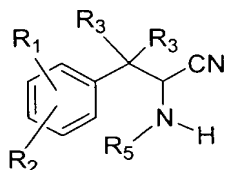
a) treating a nitrile of the formula:



with a reducing agent followed by acid hydrolysis to obtain an aldehyde of the formula:

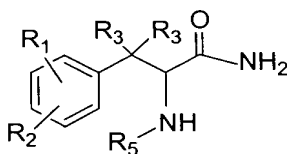


- b) reacting the aldehyde (step a) with alkali metal cyanide in the presence of an alkyl amine of the formula R_5NH_2 to obtain nitrile of the formula:



5

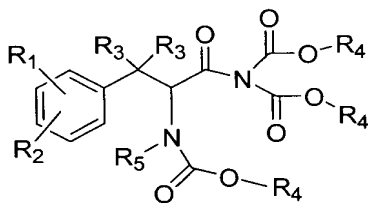
- c) hydrolyzing the nitrile (step b) with an alkali metal hydroxide to give an amide of the formula:



10

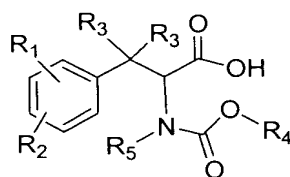
- d) reacting the amide (step c) with an organic carbonate, of the formula $O[CO_2R_4]_2$ wherein each R_4 is taken independently in the presence of dimethylaminopyridine (DMAP) to obtain a triblocked amide of the formula:

15



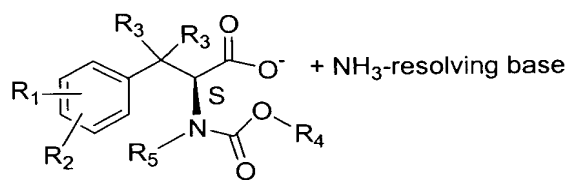
- e) hydrolyzing triblocked amide (step d) with an alkali metal hydroxide to give racemic blocked amine of the formula:

20



- f) reacting racemic blocked amine (step e) with a resolving base (NH_2 -resolving base) to obtain a resolved blocked amine salt of the formula:

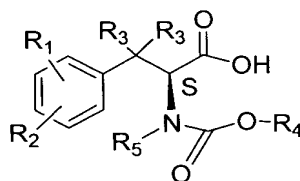
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and

- g) treating the resolved blocked amine salt with aqueous alkali metal hydroxide and acidifying with acid to give a compound of Formula I:

10



Formula I

15

2. The process according to claim 1, wherein the reducing agent in step a is tin chloride/hydrochloric acid, lithium aluminum hydride, or diisobutylaluminum hydride (DIBAL-H).

20

3. The process according to claim 2, wherein the reducing agent is diisobutylaluminum hydride (DIBAL-H).

4. The process according to claim 1, wherein the alkali metal cyanide in step b is sodium cyanide, potassium cyanide or lithium cyanide.

5. The process according to claim 4, wherein the alkali metal cyanide is potassium cyanide.
- 5 6. The process according to claim 1, wherein the alkylamine is methylamine.
7. The process according to claim 1, wherein the alkali metal hydroxide in step c is sodium hydroxide, potassium hydroxide or lithium hydroxide.
- 10 8. The process according to claim 7, wherein the alkali metal hydroxide is lithium hydroxide.
9. The process according to claim 7, optionally including the oxidizing agent hydrogen peroxide in step c.
- 15 10. The process according to claim 1, wherein the organic carbonate in step d is t-butyl dicarbonate.
11. The process according to claim 1, step d, optionally including the organic base N,N-diisopropylethylamine in step d.
- 20 12. The process according to claim 1 wherein the alkali metal hydroxide in step e is sodium hydroxide, potassium hydroxide or lithium hydroxide.
- 25 13. The process according to claim 12, wherein the alkali metal hydroxide is sodium hydroxide.
14. The process according to claim 1 wherein the resolving base in step f is (S)-(-)- α -methylbenzylamine, (S)-(-)- α -methyl-4-nitro-benzylamine, (1R,2S)-(-)-norephedrin, or (1S,2R)-(+)-norephedrin.
- 30 15. The process according to claim 14, wherein the resolving base is (S)-(α)-methylbenzylamine or (S)-(-)-methyl-4-nitro-benzylamine.

16. The process according to claim 15, wherein the resolving base is (S)-(-)- α -methylbenzylamine.

5 17. The process according to claim 1, wherein the alkali metal hydroxide in step g is sodium hydroxide, potassium hydroxide, lithium hydroxide or sodium carbonate.

18. The process according to claim 17, wherein the alkali metal hydroxide is sodium hydroxide.

10

19. The process according to claim 1, wherein the acids hydrochloric, sulfuric or citric acid in step g adjust the pH to 6 or less.

20. The process according to claim 19, wherein the pH is adjusted to 3 to 6.

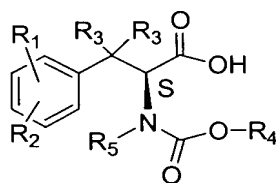
15

21. The process according to claim 20, wherein the pH is adjusted to 4 to 6.

22. The process according to claim 21 wherein the pH is adjusted to 5 to 6 with citric acid.

20

23. A process for the preparation of a compound of the formula:



Formula I

25

wherein:

R_1 and R_2 are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

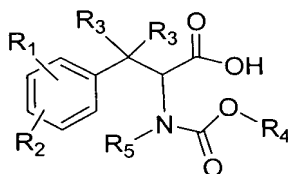
30 R_3 is straight chain alkyl of 1 to 4 carbon atoms;

R₄ is straight chain alkyl of 1 to 4 carbon atoms and branched chain alkyl of 3 to 4 carbon atoms;

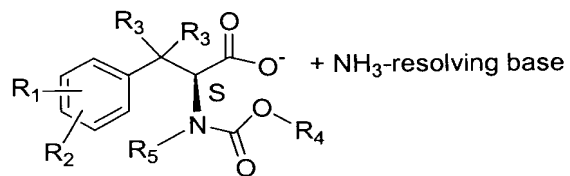
5 R₅ is straight chain alkyl of 1 to 4 carbon atoms;

comprising:

10 a) reacting a racemic blocked amine of the formula:



15 with a resolving base (NH₂-resolving base) to obtain a resolved blocked amine salt of the formula:

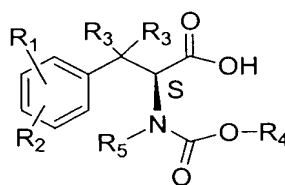


20

and

b) treating the resolved blocked amine salt with aqueous alkali metal hydroxide and acidifying with acid to give a compound of Formula I

25



Formula I

5 24. The process according to claim 23 wherein the resolving base in step a is (S)-(-)- α -methylbenzylamine, (S)-(-)- α -methyl-4-nitro-benzylamine, (1R,2S)-(-)-norephedrin, or (1S,2R)-(+)-norephedrin.

25. The process according to claim 24, wherein the resolving base is (S)-(-)- α -
10 methylbenzylamine or (S)-(-)- α -methyl-4-nitro-benzylamine.

26. The process according to claim 25, wherein the resolving base is (S)-(-)- α -methylbenzylamine.

15 27. The process according to claim 23, wherein the alkali metal hydroxide in step b is sodium hydroxide, potassium hydroxide, lithium hydroxide or sodium carbonate.

28. The process according to claim 27, wherein the alkali metal hydroxide is sodium
20 hydroxide.

29. The process according to claim 23, wherein the acids hydrochloric, sulfuric or citric acid in step h adjust the pH to 6 or less.

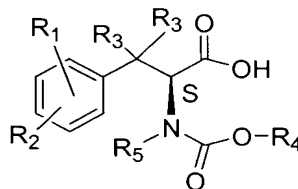
30. The process according to claim 29, wherein the pH is adjusted to 3 to 6.

25 31. The process according to claim 30, wherein the pH is adjusted to 4 to 6.

32. The process according to claim 31 wherein the pH is adjusted to 5 to 6 with citric
acid.

30

33. A process for the preparation of a compound of Formula I:



Formula I

wherein:

R_1 and R_2 are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

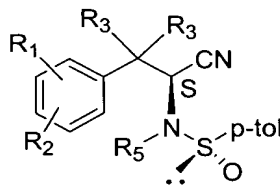
R_3 is straight chain alkyl of 1 to 4 carbon atoms;

R_4 is straight chain alkyl of 1 to 4 carbon atoms and branched chain alkyl of 3 to 4 carbon atoms;

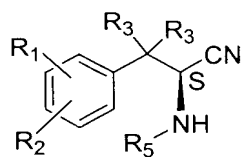
R_5 is straight chain alkyl of 1 to 4 carbon atoms;

comprising:

a) deblocking substituted amine of the formula:

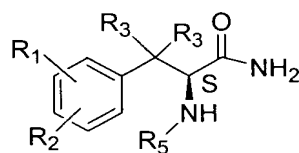


with acid to give nitrile of the formula:

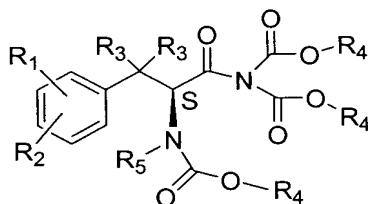


b) hydrolyzing the nitrile (step a) in the presence of an alkali metal hydroxide to obtain an amide of the formula:

5

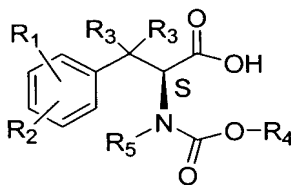


10 c) reacting the amide (step b) with an organic carbonate of the formula $O[CO_2R_4]_2$ wherein R_4 is taken independently in the presence of dimethylaminopyridine (DMAP) to give triblocked amide of the formula:



15 and;

d) hydrolyzing triblocked amide (step c) with an alkali metal hydroxide and acidifying with acid to obtain a compound of Formula I:

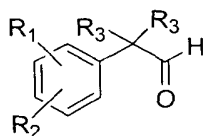


Formula I

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34. The process according to claim 33 step a wherein the acid is methanolic HCl.
35. The process according to claim 33 step b wherein the alkali metal hydroxide in step b is sodium hydroxide, potassium hydroxide or lithium hydroxide.
- 5 36. The process according to claim 35 wherein the alkali metal hydroxide is lithium hydroxide.
37. The process according to claim 36 including an optionally added oxidizing agent
10 which is hydrogen peroxide.
38. The process according to claim 33, wherein the organic carbonate in step c is t-butyl dicarbonate.
- 15 39. The process according to claim 38 optionally including an organic base in step c which is N,N-diisopropylethylamine.
40. The process according to claim 33 wherein the alkali metal hydroxide in step d is sodium hydroxide, potassium hydroxide or lithium hydroxide.
- 20 41. The process according to claim 40, wherein the alkali metal hydroxide is sodium hydroxide.
42. The process according to claim 33, step d wherein the acids hydrochloric,
25 sulfuric or citric acid in step d adjust the pH to 6 or less.
43. The process according to claim 42, wherein the pH is adjusted to 3 to 6.
44. The process according to claim 43, wherein the pH is adjusted to 4 to 6.
- 30 45. The process according to claim 44 wherein the pH is adjusted to 5 to 6 with citric acid.

46. A process for the preparation of an aldehyde of the formula:

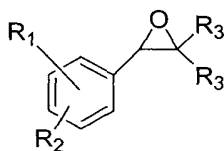


5 wherein:

R_1 and R_2 are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

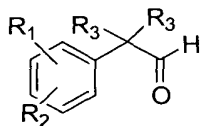
10 R_3 is straight chain alkyl of 1 to 4 carbon atoms;

by reacting a substituted oxirane of the formula:



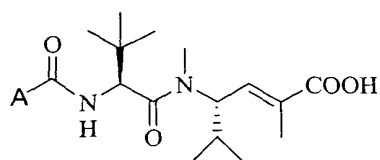
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with *tris*(pentafluorophenyl) borane to afford an aldehyde of the formula



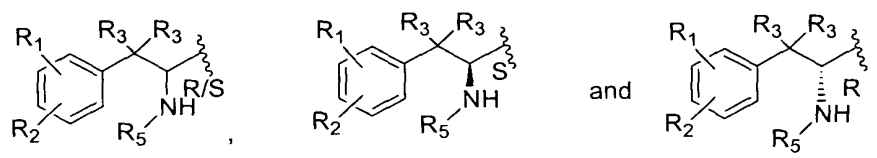
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47. A process for the preparation of a carboxylic acid of the formula:



wherein:

- 5 A is selected from the group



R₁ and R₂ are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

10

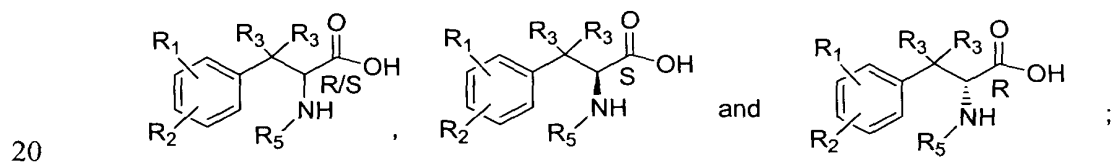
R₃ is straight chain alkyl of 1 to 4 carbon atoms;

R₅ is straight chain alkyl of 1 to 4 carbon atoms;

15

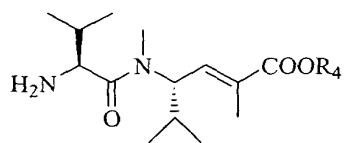
comprising:

- a) coupling an amino acid of the formula selected from the group:



20

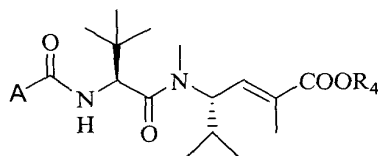
with an ester of the formula:



R₄ is straight chain alkyl of 1 to 4 carbon atoms and branched chain alkyl of 3 to 4 carbon atoms;

5

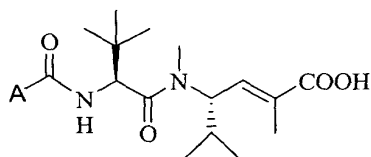
in the presence of a coupling agent and an organic base to obtain an ester of the formula:



10

b) hydrolyzing the ester of step a) with an alkali metal hydroxide and acidifying with acid affords a carboxylic acid of the formula:

15



48. A process according to claim 47 wherein the coupling agent is selected from the group N,N'-dicyclohexylcarbodiimide plus 1-hydroxybenzotriazole, benzotriazol-1-yloxytris(dimethylamino)phosphonium hexafluorophosphate (BOP-reagent), N,N'-bis[2-oxo-3-oxazolidinyl]phosphorodiamidic chloride (BOB Cl), diphenylphosphinyl chloride (DPP-Cl), diethoxyphosphoryl cyanide, 2-chloro-1-methylpyridinium iodide, phenyldichlorophosphate plus imidazole, 2-(1H-benzotriazole-1-yl)-1,1,3,3-tetramethyluronium tetrafluoroborate, 2-(1H-

benzotriazole-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate, bromo-tris-pyrrolidino-phosphonium hexafluorophosphate and benzotriazole-1-yl-oxy-tris-pyrrolidino-phosphonium hexafluorophosphate in the presence of N,N-diisopropylethylamine.

5

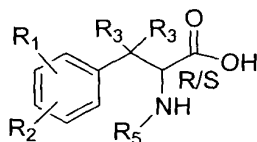
49. A process according to claim 48 wherein the coupling agent is benzotriazole-1-yl-oxy-tris-pyrrolidino-phosphonium hexafluorophosphate.

50. A process according to claim 47 wherein the organic base is N,N-diisopropylethylamine, N-methylmorpholine, N-methylpyrrolidine, triethylamine, 4-dimethylaminopyridine, 2,6-di-tert-butyl-4-methylpyridine or pyridine.

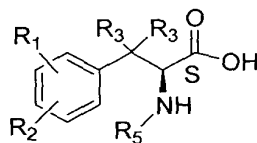
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51. The process according to claim 47 wherein the aminoacid is

15

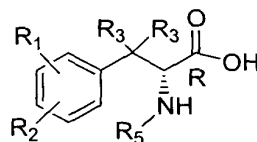


52. The process according to claim 47 wherein the aminoacid is



53. The process according to claim 47 wherein the aminoacid is

20



54. A process according to claim 50 wherein the organic base is N,N-diisopropylethylamine.

25

55. A process according to claim 47 wherein the alkali metal hydroxide is sodium hydroxide, potassium hydroxide or lithium hydroxide.

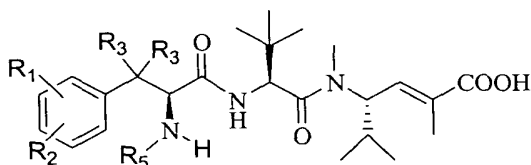
5 56. A process according to claim 55 wherein the alkali metal hydroxide is lithium hydroxide.

57. A process according to claim 47 wherein the acid is selected from sulfuric, hydrochloric or citric.

10

58. A process for the preparation of a carboxylic acid of the formula:

6



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wherein:

R₁ and R₂ are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

20

R₃ is straight chain alkyl of 1 to 4 carbon atoms;

R₄ is straight chain alkyl of 1 to 4 carbon atoms and branched chain alkyl of 3 to 4 carbon atoms;

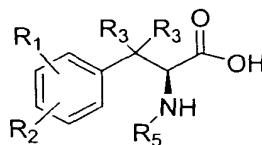
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R₅ is straight chain alkyl of 1 to 3 carbon atoms;

comprising:

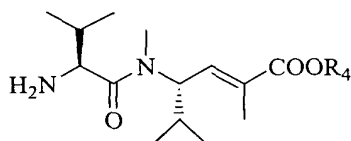
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a) coupling an amino acid of the formula:



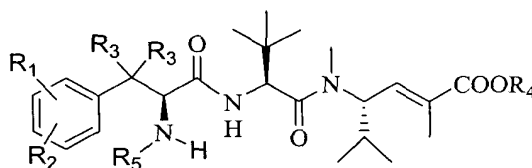
with an ester of the formula:

5

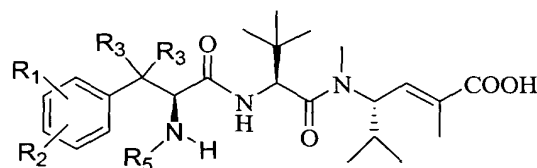


in the presence of a coupling agent and an organic base to obtain an ester of the formula:

10



15 b) hydrolyzing the ester of step a) with an alkali metal hydroxide and acidifying with acid to obtain a carboxylic acid of the formula:



20 59. A process according to claim 58 wherein the coupling agent is selected from the group N,N'-dicyclohexylcarbodiimide plus 1-hydroxybenzotriazole, benzotriazol-1-yloxytris(dimethylamino)phosphonium hexafluorophosphate (BOP-reagent), N,N'-bis[2-oxo-3-oxazolidinyl]phosphorodiamidic chloride (BOB Cl),

diphenylphosphinyl chloride (DPP-Cl), diethoxyphosphoryl cyanide, 2-chloro-1-methylpyridinium iodide, phenyldichlorophosphate plus imidazole, 2-(1H-benzotriazole-1-yl)-1,1,3,3-tetramethyluronium tetrafluoroborate, 2-(1H-benzotriazole-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate, bromo-tris-pyrrolidino-phosphonium hexafluorophosphate and benzotriazole-1-yl-oxy-tris-pyrrolidino-phosphonium hexafluorophosphate in the presence of N,N-diisopropylethylamine.

60. A process according to claim 59 wherein the coupling agent is benzotriazole-1-yl-oxy-tris-pyrrolidino-phosphonium hexafluorophosphate.

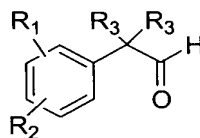
61. A process according to claim 58 wherein the organic base is N,N-diisopropylethylamine, N-methylmorpholine, N-methylpyrrolidine, triethylamine, 4-dimethylaminopyridine, 2,6-di-tert-butyl-4-methylpyridine or pyridine.

62. A process according to claim 61 wherein the organic base is N,N-diisopropylethylamine.

63. A process according to claim 58 wherein the alkali metal hydroxide is sodium hydroxide, potassium hydroxide or lithium hydroxide.

64. A process according to claim 63 wherein the alkali metal hydroxide is lithium hydroxide.

65. A process for the preparation of an aldehyde of the formula:



wherein:

R_1 and R_2 are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

R_3 is straight chain alkyl of 1 to 4 carbon atoms;

5

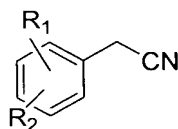
R_4 is straight chain alkyl of 1 to 4 carbon atoms and branched chain alkyl of 3 to 4 carbon atoms;

R_5 is straight chain alkyl of 1 to 4 carbon atoms;

10

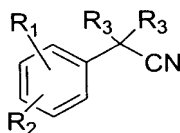
comprising:

a) alkylating benzylnitrile of the formula:



15

with an alkylating agent $R_3\text{LG}$, in the presence of a strong base, to obtain a nitrile of the formula:

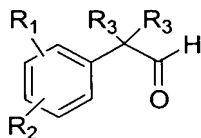


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and

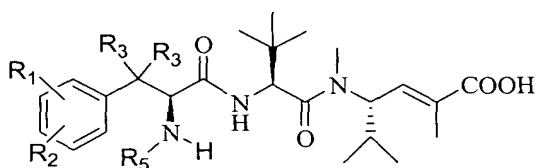
b) reducing a nitrile of step a with a reducing agent to give an aldehyde of the formula:

25



66. A process according to claim 65 wherein the strong base according to claim step a is potassium *tert*-butoxide (^tBuOK), sodium *tert*-butoxide (^tBuONa), lithium *tert*-butoxide (^tBuOLi), sodium hydride, potassium hydride, sodium hydroxide, or lithium diisopropylamide.
67. A process according to claim 66 wherein the strong base is potassium *tert*-butoxide (^tBuOK).
68. A process according to claim 65 step a wherein the alkylating agent R₃LG is methyl iodide or methyl bromide or optionally methyl bromide in the presence of sodium iodide or tetrabutyl ammonium iodide.
69. A process according to claim 65 step b wherein the reducing agent is tin chloride/hydrochloric acid, lithium aluminum hydride or diisobutylaluminum hydride (DIBAL-H).
70. A process according to claim 69 wherein the reducing agent is diisobutylaluminum hydride (DIBAL-H).
71. The compound which is 3-methyl-2-(methylamino)-3-phenylbutanenitrile.
72. The compound which is N,β,β-trimethylphenylalaninamide.
73. The compound which is N,β,β-trimethylphenylalanine.
74. The compound which is N-(*tert*-butoxycarbonyl)-N,β,β-trimethylphenylalaninamide.
75. The compound which is N,N,N-tris(*tert*-butoxycarbonyl)-N,β,β-trimethylphenylalaninamide.
76. The compound which is N-(*tert*-butoxycarbonyl)-N,β,β-trimethylphenylalanine.

77. The compound which is (S)-N-(2-methyl-2-phenyl-propylidene)-p-toluene-sulfonamide.
- 5 78. The compound which is (S_S,2S)-N-(p-toluenesulfinyl)-2-amino-3-methyl-3-phenylbutyronitrile.
79. The compound which is (S_S,2R)-N-(p-toluenesulfinyl)-2-amino-3-methyl-3-phenylbutyronitrile.
- 10 80. The compound which is (S_S,2S)-N-(p-toluenesulfinamido-methyl)-3-methyl-3-phenylbutanenitrile.
81. The compound which is (2S)-3-methyl-2-(methylamino)-3-phenylbutanenitrile,
15 N,β,β-trimethyl-L-phenylalaninamide.
82. The compound which is N-(tert-butoxycarbonyl)-N,β,β-trimethyl-L-phenylalaninamide.
- 20 83. The compound which is N,N,N-tris(tert-butoxycarbonyl)-N,β,β-trimethyl-L-phenylalaninamide.
84. The compound which is salt of N-(tert-butoxycarbonyl)-N,β,β-trimethyl-L-phenylalanine with (S)-(-)-α-methylbenzylamine (SAMBA).
- 25 85. The compound which is salt of N-(tert-butoxycarbonyl)-N,β,β-trimethyl-D-phenylalanine with (R)-(+)-α-methylbenzylamine (RAMBA).
- 30 86. A process for the preparation of a carboxylic acid of the formula:



wherein:

- 5 R_1 and R_2 are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

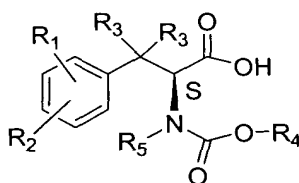
R_3 is straight chain alkyl of 1 to 4 carbon atoms;

- 10 R_4 is straight chain alkyl of 1 to 4 carbon atoms and branched chain alkyl of 3 to 4 carbon atoms;

R_5 is straight chain alkyl of 1 to 3 carbon atoms;

- 15 comprising:

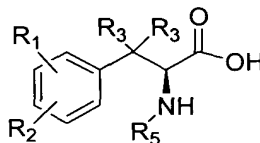
a) reacting a compound of Formula I:



20

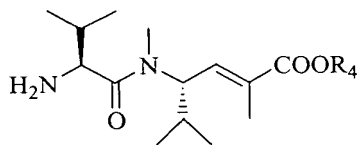
Formula I

With acid to obtain an amino acid of the formula:

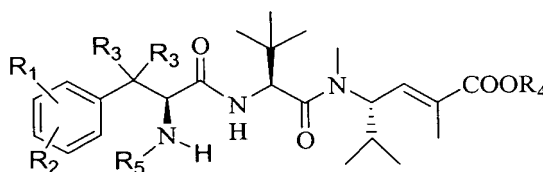


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b) coupling an amino acid of step a with an ester of the formula:

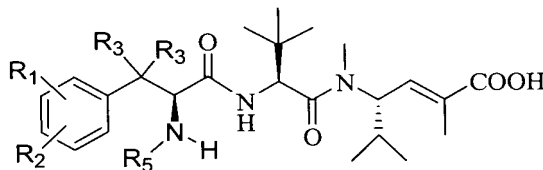


5 in the presence of a coupling agent and an organic base to obtain an ester of the formula:



10

c) hydrolyzing the ester of step b) with an alkali metal hydroxide and acidifying with acid to obtain a carboxylic acid of the formula:



15

87. A process according to claim 86 wherein the coupling agent is selected from the group N,N'-dicyclohexylcarbodiimide plus 1-hydroxybenzotriazole, benzotriazol-1-
 20 yloxytris(dimethylamino)phosphonium hexafluorophosphate (BOP-reagent),
 N,N'-bis[2-oxo-3-oxazolidinyl]phosphorodiamidic chloride (BOB Cl),
 diphenylphosphinyl chloride (DPP-Cl), diethoxyphosphoryl cyanide, 2-chloro-1-
 methylpyridinium iodide, phenyldichlorophosphate plus imidazole, 2-(1H-
 benzotriazole-1-yl)-1,1,3,3-tetramethyluronium tetrafluoroborate, 2-(1H-
 benzotriazole-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate, bromo-tris-
 25 pyrrolidino-phosphonium hexafluorophosphate and benzotriazole-1-yl-oxy-tris-

pyrrolidino-phosphonium hexafluorophosphate in the presence of N,N-diisopropylethylamine.

88. A process according to claim 87 wherein the coupling agent is benzotriazole-1-yl-oxy-tris-pyrrolidino-phosphonium hexafluorophosphate.

89. A process according to claim 86 wherein the organic base is N,N-diisopropylethylamine, N-methylmorpholine, N-methylpyrrolidine, triethylamine, 4-dimethylaminopyridine, 2,6-di-tert-butyl-4-methylpyridine or pyridine.

10

90. A process according to claim 89 wherein the organic base is N,N-diisopropylethylamine.

91. A process according to claim 86 wherein the alkali metal hydroxide is sodium hydroxide, potassium hydroxide or lithium hydroxide.

15

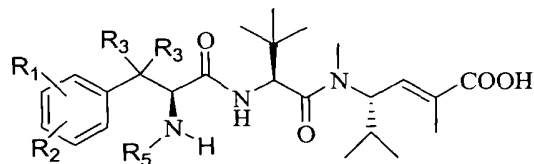
92. A process according to claim 91 wherein the alkali metal hydroxide is lithium hydroxide.

93. A process according to claim 86 step a wherein the acid is trifluoroacetic acid.

20

94. A process for the preparation of a carboxylic acid of the formula:

74



25 wherein:

R₁ and R₂ are independently selected from H, straight chain alkyl of 1 to 4 carbon atoms, halogen and alkoxy of 1 to 4 carbon atoms;

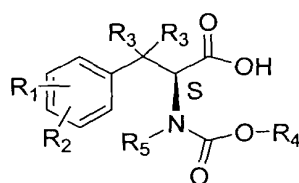
30 R₃ is straight chain alkyl of 1 to 4 carbon atoms;

R₄ is straight chain alkyl of 1 to 4 carbon atoms and branched chain alkyl of 3 to 4 carbon atoms;

5 R₅ is straight chain alkyl of 1 to 3 carbon atoms;

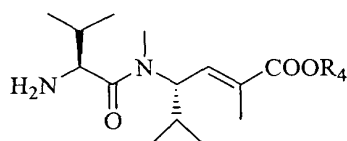
comprising:

10 a) coupling a compound of Formula I:

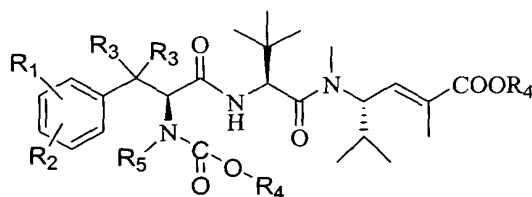


Formula I

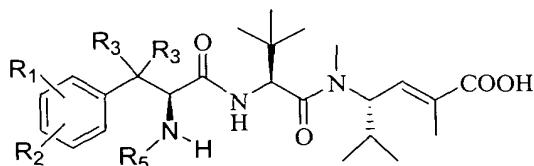
15 with an ester of the formula:



20 in the presence of a coupling agent and an organic base to obtain an ester of the formula:



b) hydrolyzing the ester of step a) with an alkali metal hydroxide and treating with acid to obtain a carboxylic acid of the formula:



5

95. A process according to claim 94 wherein the coupling agent is selected from the group N,N'-dicyclohexylcarbodiimide plus 1-hydroxybenzotriazole, benzotriazol-1-yl-oxytris(dimethylamino)phosphonium hexafluorophosphate (BOP-reagent),
 10 N,N'-bis[2-oxo-3-oxazolidinyl]phosphorodiamidic chloride (BOB Cl),
 diphenylphosphinyl chloride (DPP-Cl), diethoxyphosphoryl cyanide, 2-chloro-1-methylpyridinium iodide, phenyldichlorophosphate plus imidazole, 2-(1H-benzotriazole-1-yl)-1,1,3,3-tetramethyluronium tetrafluoroborate, 2-(1H-benzotriazole-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate, bromo-tris-pyrrolidino-phosphonium hexafluorophosphate and benzotriazole-1-yl-oxy-tris-pyrrolidino-phosphonium hexafluorophosphate in the presence of N,N-diisopropylethylamine.

96. A process according to claim 95 wherein the coupling agent is benzotriazole-1-yl-oxy-tris-pyrrolidino-phosphonium hexafluorophosphate.

97. A process according to claim 94 wherein the organic base is N,N-diisopropylethylamine, N-methylmorpholine, N-methylpyrrolidine, triethylamine, 4-dimethylaminopyridine, 2,6-di-tert-butyl-4-methylpyridine or pyridine.

25

98. A process according to claim 97 wherein the organic base is N,N-diisopropylethylamine.

99. A process according to claim 94 wherein the alkali metal hydroxide is sodium hydroxide, potassium hydroxide or lithium hydroxide.

30

100. A process according to claim 99 wherein the alkali metal hydroxide is lithium hydroxide.

5 101. A process according to claim 94 step a wherein the acid is trifluoroacetic acid.